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Note

Recommendations for reporting thermal analysis data

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The original recommendations for reporting thermal analysis data was developed in the late 1960s and early 1970s by the sterling efforts of McAdie [1–3]. In 1991, it was decided by the Standardization Committee to review and revise the recommendations to take into account the changes that had taken place in the intervening 20 years. The first focus group was convened in the Department of Chemistry at the University of Toledo in 1992, where I was spending a sabbatical working with the late David Dollimore. The group consisted of Roger Blaine, Bruce Cassel, David Dollimore, Joe and Rosemary Flynn, Pat Gallagher, John Kelly, Harry McAdie, Don Melotik and Mike Neag. The findings of this group were presented at the ICTAC Conference in London in 1992, and published in 1993 [4].

Subsequently various draft proposals were circulated for comment to the 16 members of the Standardization Committee, revised accordingly, and presented to the Standardization Committee Chairman for consideration by ICTAC Council in early 1997. Since that time, some minor modifications have been made. At the ICTAC Conference held in Copenhagen in 2000, it was agreed by the President of ICTAC, Dr. Jean Rouquerol, and the Chairman of the Scientific Commission, Prof. Eberhard Gmelin, that if no further comments were received by the end of 2001 that the recommendations should be published.

The revised version builds on the recommendations produced by McAdie [1–3]. However, given the large amount of experimental data reported in the literature on the importance of the sample properties to the thermal analysis result, the section on sample properties has been separated out and expanded. We hope that this stresses that defining the properties of the sample is of similar importance to defining the experimental variables in obtaining reproducible and explainable results. The replacement of the chart recorder by the computer is also recognized by the addition of a section on what aspects of data acquisition and data treatment might be noted to assist the reader.

1. Recommendations for reporting thermal analysis data

1.1. Scope

This practice is for general use in reporting experimental information from differential thermal analysis, thermogravimetry, evolved gas analysis or detection, and thermomechanical analysis studies. Accurate and comprehensive reporting of experimental conditions is required, since changes in the conditions may produce changes in the results obtained. Reporting in full the following information will enable another worker to reproduce the experiment, or to reconcile differences that may be apparent in another study:

- Properties of the substances used in the experiments, with special attention to the sample under investigation.

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- Experimental parameters used to carry out the experiments.
- Data acquisition techniques and data treatment used to produce the final printout of the results.

This paper does not address safety problems associated with the use of the related materials or equipment. It is the responsibility of the user to establish the appropriate safety standards and work within the validity of the given safety laws.

2. Information required for all thermal analysis techniques

Accompanying each thermal analysis record should be information about the following topics.

2.1. Properties of substances

1. Identification of all substances (sample and, if used, reference and/or diluent) by a definitive name, an empirical formula, or equivalent compositional information.
2. A statement of the source of all substances, details of their histories, pretreatments, physical properties, and chemical purities, as far as they are known.
   - 2.1. ''Histories'' includes method of acquisition or manufacture of the sample (how it was isolated or made, manufacturing conditions, e.g. grinding, sizing, etc.), previous thermal and mechanical treatments (e.g. repeated grinding, sintering, all experimental conditions), surface modification, and any other physical or chemical treatment.
   - 2.2. ''Pretreatments'' includes preparation (e.g. compaction) of the sample prior to the thermal analysis experiment.
   - 2.3. ''Physical properties'' includes particle size, surface area, porosity.
   - 2.4. ''Chemical purities'' includes elemental analysis, phase composition, and chemical homogeneity of sample.

2.2. Experimental conditions

1 Identification of the apparatus, by the provision of the manufacturer's name and model number. More detail should be provided if significant modifications to the standard commercial model have been made, or if the apparatus is new or novel, or if the apparatus is not commercially available.
2 Sample mass geometry and dimensions.
3 A statement of the dimensions, geometry and materials of the sample holder pan, and where applicable the method of loading the sample into the pan.
4 Identification of the sample atmosphere by pressure, composition, and purity: whether the atmosphere is static, self-generated or dynamic through or over the sample. Where applicable, the ambient atmospheric pressure and humidity should be specified. If the pressure is other than atmospheric, full details of the method of control should be given.
5 A clear statement of the temperature environment of the sample during measurement or reaction, including initial temperature, final temperature, rate of temperature change if linear, or details if not linear.
6 Identification of the abscissa scale in terms of time or temperature at a specified location. Time or temperature should be plotted to increase from left to right.
7 A statement of the methods used to identify intermediate or final products.
8 Faithful reproductions of all original records.
9 Wherever possible, each thermal event should be identified and supplementary supporting evidence stated.

2.3. Data acquisition and manipulation methods

1 Identification of the instrument manufacturers software version, or details of any self-developed versions.
2 Equations used to process data, or reference to suitable literature.
3 The frequency of sampling, filtering and averaging of the signal.
4 An indication of the smoothing and signal conditioning used to convert analogue to digital signals, or reference to suitable literature. Similar information for subsequent processing of the digital data is also needed.

3. Information required for specific thermal analysis techniques

3.1. Differential thermal analysis (DTA) and differential scanning calorimetry (DSC)

1 The ordinate scale should indicate the temperature differential (DTA) or heat flow (DSC) at a specific temperature (or time). Exothermic and endothermic directions should be clearly marked.
2 The method and materials used for the calibration of temperature, enthalpy and specific heat capacity.
3 For temperature modulated DSC, the amplitude and frequency of modulation and the type of waveform.

3.2. Thermogravimetry (TG)

1 The ordinate scale should indicate the mass scale. Mass loss should be plotted as a downward trend and deviations from this practice should be clearly marked. Additional scales (for example, fractional decomposition, molecular composition) may be used for the ordinate where desired.
2 The method and materials used for the calibration of the temperature and mass.
3 If derivative thermogravimetry is employed, the method of obtaining the derivative should be indicated and the units of the ordinate specified.

3.3. Evolved gas analysis (EGA)

1 Identification of the ordinate scale in specific terms where possible. In general, increasing concentrations of evolved gas should be plotted upwards. For gas density detectors, increasing gas density should also be plotted upwards. Deviations from these practices should be clearly marked.
2 If not a standard manufactured system, the flow rate, total volume, construction, method of heating, and temperature of the interface between the thermal analysis apparatus and the detector should be given, together with an estimate of the time delay within the system.
3 Identification of the gas detector used.

3.4. Thermomechanical analysis (TMA)

1 A statement of the type of deformation (tensile, torsional, bending etc.) and the dimensions, geometry and materials of the loading elements.
2 The method and materials for the calibration of temperature and extent of deformation, where applicable.
3 Identification of the ordinate scale in specific terms where possible. For static procedures, increasing expansion or extension, and torsional displacement should be plotted upwards. Increased penetration or deformation in flexure should be plotted downwards.

References

[1] H.G. McAdie, Recommendations for reporting thermal analysis data, Anal. Chem. 39 (1967) 543.
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[3] H.G. McAdie, Recommendations for reporting thermal analysis data. Thermomechanical techniques, Anal. Chem. 46 (8) (1974) 1146–1147.
[4] J.G. Dunn, Recommendations for reporting thermal analysis data, J. Therm. Anal. 40 (1992) 1431–1436.